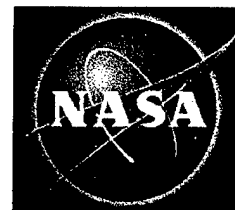


# NEW DEVELOPMENTS IN HIGH TEMPERATURE CERAMICS

AUGUST 12 - 15, 1998  
ISTANBUL / TURKEY

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Time	WEDNESDAY Aug 12, 98	THURSDAY Aug 13, 98	FRIDAY Aug 14, 98	SATURDAY Aug 15, 98
8.00 - 8.30	Ali Sayir & S. C. Farmer N. Claussen - T. Baykara	M. Birkan / USA	D. B. Marshall / USA	S. M. Wiederhorn / USA
8.30 - 9.00	B. Derby / UK	M. Opeka / USA	W. T. Petuskey / USA	A. J. Ardell / USA
9.00 - 9.30	E. Gutmanas / Israel	M. D. Sacks / USA	B.Saruhan / Germany	V. Tikare / USA
9.30 -10.00	R. Pampuch / Poland	R. E. Tressler / USA	A.N. Cormack /USA	R. K. Kalia / USA
10.00-10.30	C O F F E E B R E A K			
10.30-11.00	F. Thevenot / France	M. Hoffmann / Germany	A. Glaeser / USA	P. Vashista / USA
11.00-11.30	F. Aldinger / Germany	H. T. Larker / Sweden	S. Pennycook / USA	C.-K. Loong / USA
11.30-12.00	F. Cambier / Belgium	I-Wei Chen / USA	D. J. Green / USA	M. Opeka / USA
12.00-12.20	N. Claussen / Germany	H. Mandal / Turkey	E. Dickey / USA	P. Descamp / Belgium
12.20-12.40	M. Akinc / USA	N. Durlu /Turkey	D. L. Chubb / USA	M. Gulgun / Germany
12.40-13.00	L U N C H	R. Janssen / Germany	L U N C H	A. Sayir / USA
13.00-13.30		L U N C H		L U N C H
13.30-14.00		Bus Departs 14.00		Bus Departs 14.00
14.00-14.30	TOPKAPI  PALAST	BLUE MOSQUE  and HAGIA SOPHIA	H. Schneider / Germany	BOSPHORUS TOUR
14.30-15.00			W. Kriven / USA	
15.00-15.30			I. Aksay /USA	
15.30-16.00			R. Weber / USA	
16.00-16.30	16.30-18.00	Return to Conrad Int.	L. E. Matson / USA	BANQUETT DINNER starts 18.30
16.30-18.00			POSTERS: F.Kara, Anadolu Uni. -- S. Turan* Anadolu Uni. N.O. Engin et.al./ODTU -- W. M. Kriven / U. Illinois D. Bayraktar et.al./ODTU -- E. Suvaci, et.al. / Penn State M. Shapiro et. al. /Technion -- N. Durlu / TUBITAK I. E. Gönenli, et. al. /ODTU -- H.Mandal /U. Karlsruhe	
18.00-18.30	Return to Conrad Int.	Return to Conrad Int.		

# WEDNESDAY

August 12, 1998

## NEW PROCESSING ROUTES: Prof. N. Claussen - TUHH

8.00 - 8.30 Introductory Remarks - Welcome

A. Sayir and S. C. Farmer - NASA / CWRU

N. Claussen - TUHH

T. Baykara - TUBITAK

8.30 - 9.00 New Developments in High Temperature Reaction Formed Ceramics

**B. Derby\*** - University of Oxford - UK

9.00 - 9.30 High Temperature Ceramics by Thermal Explosion Reactions

**E. Y. Gutmanas\*** and I. Gotman - Technion/Israel Inst. of Techn. - Israel

9.30 - 10.00 Solid Combustion: An Efficient Method for Producing Advanced Powders

**R. Pampuch\*** - Department of Advanced Ceramics, AGH - Poland

## 10.00 - 10.30 COFFEE BREAK

## NEW PROCESSING ROUTES: Dr. Ali Sayir - NASA / CWRU

10.30 - 11.00 Preparation, Mechanical and Microstructural Characterization, Wear Resistance of SiC - TiB<sub>2</sub> Ceramic Composites

**F. Thevenot\*** - Ecole Nationale Supérieure des Mines - France

11.00 - 11.30 Novel High Temperature Si-B-C-N Glass Ceramics

**F. Aldinger,\*** Max-Planck-Inst. für Metallforschung, Stuttgart - Germany

11.30 - 12.00 Creep Behaviour of Al<sub>2</sub>O<sub>3</sub> - SiC Nano - Composites

**F. Cambier,\*** and P. Descamps - Belgian Ceramic Research Centre - Belgium

12.00 - 12.20 Microstructure and Properties of Al-Assisted Sintered Fe/Al<sub>2</sub>O<sub>3</sub> Cermets

S. Schicker, T. Erny, D. E. Garcia, R. Janssen, and **N. Claussen\***

Adv. Ceram. Group, Techn. Univ. Hamburg, Harburg - Germany

12.20 - 12.40 Oxidation and Creep Resistant Mo-Si-B Intermetallics For High Temperature Structural Applications

**Mufit Akinc,\*** Dept. of Mat. Sci. & Eng. and Ames Lab., ISU, Ames IA - USA

## 12.40 - 14.00 LUNCH

14.00 - Bus Departure to *Topkapi Palast*

18.30 - 19.00 Return to Conrad International Istanbul

## NEW DEVELOPMENTS IN HIGH TEMPERATURE REACTION FORMED CERAMICS

Brian Derby\* and L. J. Zhao  
Department of Materials, University of  
Oxford,  
Parks Rd., Oxford, OX1 3PH, UK.

Reaction formed oxide ceramics were developed a number of years ago using the combined oxidation and sintering of Al/Al<sub>2</sub>O<sub>3</sub> green compacts and are attractive as near-net-shape manufacture is possible in principle. Zero shrinkage is possible for a range of green body densities which depend on the relative volume expansion and volume fraction of the oxidising component. Very large volume expansions occur with the formation of mullite and mullite is an interesting material for a number of applications because of its low coefficient of thermal expansion and good thermal shock resistance. The formation of mullite from siloxane polymer/Al metal preforms will be reported. Studies of phase and microstructure evolution show the importance of SiO formation as an intermediate phase with implications for loss of Si from the component during processing.

## HIGH TEMPERATURE CERAMICS BY THERMAL EXPLOSION REACTIONS

Elazar Y. Gutmanas\* and Irena Gotman  
Department of Materials Engineering,  
Technion, Haifa, 32000, Israel

Near fully dense *in situ* ceramic matrix composites were fabricated from blends of fine Ti-B<sub>4</sub>C, Ti-BN, Ti-SiC, Ti-B<sub>6</sub>Si, and Al-TiO<sub>2</sub> powders without or with the addition of Ni. Two reactive synthesis techniques were employed: 1) thermal explosion/TE (SHS) under pressure, when the compact was placed and heated from the punches and walls of the die heated slightly above the ignition temperature and 2) reactive hot pressing/RHP. In both approaches, the processing or preheating temperature ( $\leq 1100$  °C) was considerably lower than those typical of current methods used for processing of ceramic matrix composites. Partial to full conversion of reagents into products was achieved during TE, and a moderate external pressure of 150 MPa was sufficient to ensure full density of the final products. Since the die acts as a heat sink after thermal explosion rapid cooling of the product takes place resulting in fine/micron size microstructure RHP processing yielded dense materials with even finer microstructures, however full conversion of reagents into products was not achieved.

# **SOLID COMBUSTION: AN EFFICIENT METHOD FOR PRODUCING ADVANCED POWDERS**

**Roman Pampuch\***

Department of Advanced Ceramics, Acad. of  
Science, Cracow, Poland  
al. Mickiewicza 30, 30-090 Cracow - AGH A-3  
- Poland

Optimum properties of materials for performing specified functions can be attained by appropriate phase composition and microstructural design provided high quality powders are used. Most methods developed for producing high quality powders are sophisticated and not cost-, energy- and time-efficient. The energy- and time-efficiency of powder synthesis can be improved and further processing to materials simplified when the powders are produced by solid combustion. This is especially true for single phase and multiphase composite powders based on refractory non-oxide compounds like silicon nitride and carbide and of powders of intermetallic compounds. The paper deals with production of solid combustion-derived powders of these types and their application to produce nano/micro composites, nanolaminates and materials having an interpenetrating microstructure.

# **PREPARATION, MECHANICAL AND MICROSTRUCTURAL CHARACTERIZATIONS, WEAR RESISTANCE OF SiC - TiB<sub>2</sub> CERAMIC COMPOSITES.**

**Francois Thévenot\***

Ecole Nationale Supérieure des Mines  
F- 42023 SAINT-ETIENNE Cedex 2

Dense composites SiC - TiB<sub>2</sub> (5, 10 and 15 vol% TiB<sub>2</sub>) have been by reactive pressureless sintered, starting from TiO<sub>2</sub>, B<sub>4</sub>C and a phenolic resin, used as carbon precursor, following the reaction (occurring at 1400° C).  
$$\text{TiO}_2 + 0.5 \text{B}_4\text{C} + 1.5 \text{C} \rightarrow \text{TiB}_2 + \text{CO}\uparrow$$
  
The materials have been characterized, regarding the microstructure, the mechanical and tribological properties.

The dispersion of TiB<sub>2</sub> particles is quite homogeneous, observed both by optical and scanning electron microscopies. Image analysis has revealed a majority of submicronic particles. Atomic force and transmission electron microscopies have shown the presence of nanometric particles.

Concerning mechanical properties, toughness increases with the TiB<sub>2</sub> content, whereas hardness drops when the TiB<sub>2</sub> content increases. Using a pin on disc type tribometer, composites materials are less worn than monolithic SiC. Wear mechanisms are modified both in air and in water. In air, an oxidised debris layer, protective when stable, is formed in composites, whereas there is rollers formation with SiC. In water, composites are polished, and SiC is worn by fragile ruptures (cleavages). Tribooxidation of the TiB<sub>2</sub> phase is responsible of the wear resistance of composites : in air, a lubrication by the products issued of the tribooxidation may be considered.

## NOVEL Si-B-C-N GLASS CERAMICS

Fritz Aldinger\*

Max-Planck-Institut für Metallforschung  
and

Institut für Nichtmetallische Anorganische  
Materialien, Universität Stuttgart  
Pulvermetallurgisches Laboratorium,  
Heisenbergstraße 5, 70569 Stuttgart,  
Germany

The condensation of elementorganic polymers into merely inorganic glass-type materials by solid state pyrolysis is a proper way of controlling composition, atomic array and microstructure of materials. The general idea behind this kind of materials synthesis is that the aimed atomic architecture of the inorganic material will be designed into the preceramic compound by means of common polymer synthesis routes. Another feature of this process is that shaping of products and components is done in the polymer state thus economic standard techniques of polymer process engineering can be applied for the production of ceramic materials.

For the manufacture of high temperature materials covalently bonded inorganics on the basis of Si, C, N and B are of special interest. Because of the lack of grain boundaries in the amorphous state and the inherently low atomic mobility in such materials at rather high temperature, they provide substantial thermal stability, high oxidation resistance and very attractive mechanical properties at temperatures up to 1600 °C.

## CREEP BEHAVIOUR OF $\text{Al}_2\text{O}_3$ - SiC NANO-COMPOSITES

F. Cambier\* and P. Descamps

Belgian Ceramic Research Centre  
Avenue Gouverneur Cornez 4, B 7000 Mons  
Belgium

Compared with monolithic fine-grained alumina, the  $\text{Al}_2\text{O}_3$  matrix reinforced with SiC nano-particles displays especially high modulus of rupture as well as reduced creep strain.

Taking into account the fracture mode change, the aspect of ground surface showing plastic grooving, the low sensitivity to wear and the weak erosion rate dependence with grain size, it can be reasonably assumed that the strength improvement is rather associated with an increase of the interface cohesion (due to bridging by SiC particles) than with a grain size refinement involving substructure formation (as initially suggested by Niihara).

In the present work, creep tests have been performed and the results agree with such a reinforcement of the mechanical properties as the pinning effect of SiC particles tends to hinder grain boundary sliding resulting to a large creep resistance improvement. In addition, SiC particles, while counteracting the sliding phenomenon, elastically deform, giving therefore rise to a viscoelastic contribution to creep.

As another consequence of the increase of the interface strength, the crept samples are able to support stress levels, which overcome the threshold value necessary to activate dislocation motion. The high stress exponent value as well as the presence of high dislocation density in the strained grains allow us to foresee that a lattice mechanism could take part of the deformation process. Finally, an attempt of modelling is proposed which fits well the experimental creep results.



## MICROSTRUCTURE AND PROPERTIES OF Al-ASSISTED SINTERED Fe/Al<sub>2</sub>O<sub>3</sub> CERMETS

S. Schicker, T. Erny, D. E. Garcia, R. Janssen, and N. Claussen\*  
Advanced Ceramics Group, Technische  
Universitaet Hamburg-Harburg,  
Germany

Fe/Al<sub>2</sub>O<sub>3</sub> composites with metal contents between 23 and 35 vol % have been fabricated by Al-assisted pressureless sintering. The effect of oxygen partial pressure variation during sintering on phase development, microstructure and mechanical properties has been investigated.

Formation of the spinel phase FeO•Al<sub>2</sub>O<sub>3</sub> (hercynite) was found to occur when sintering in air. Sintering in argon avoided spinel formation. The cermet microstructures exhibit interpenetrating networks. Fracture toughness increases with increasing Fe content depending strongly on the phase composition. Cermets containing small amounts of spinel show fracture toughnesses of 7.1 MPam<sup>1/2</sup>, while those sintered in Ar having no spinel reach values up to 7.1 MPam<sup>1/2</sup>. Similar Al-assisted pressureless reaction sintered Al<sub>2</sub>O<sub>3</sub> cermets were fabricated based on Nb and Cr. This new class of inexpensive and tough cermets may be suitable for various high-temperature applications.

## OXIDATION AND CREEP RESISTANT MO-SI-B INTERMETALLICS FOR HIGH TEMPERATURE STRUCTURAL APPLICATIONS

Mufit Akinc\*  
Department of Materials Science &  
Engineering and  
Ames Laboratory, Iowa State University,  
Ames IA 50011

Use of Mo<sub>5</sub>Si<sub>3</sub> in high temperature applications is limited by oxidation induced catastrophic failure above 800 °C. Oxidation resistance of Mo<sub>5</sub>Si<sub>3</sub> is dramatically improved from 800 to 1500 °C by addition of less than 2% boron. The oxidation rate is decreased by five orders of magnitude at 1200 °C by the addition of Boron. Mechanism of oxidation protection was found to be formation of a protective borosilicate layer due to viscous flow. Compressive creep rate of Mo-Si-B intermetallic compositions in the vicinity of Mo<sub>5</sub>Si<sub>3</sub>B<sub>x</sub> was found to be similar to that of undoped Mo<sub>5</sub>Si<sub>3</sub> and at least an order of magnitude lower than MoSi<sub>2</sub> promising a new family of materials

for high temperature structural applications. Creep rate at 1240 °C and at 138 MPa was measured to be 1.8x10<sup>-7</sup> s<sup>-1</sup>. Creep tests also revealed an average creep activation energy and stress component to be E = 400 kJ/mol, and n = 4.3 respectively. Implications of improvement in oxidation resistance with small boron additions to other high melting intermetallics are very intriguing. Potential candidates and challenges ahead will be discussed also.

# THURSDAY

August 13, 1998

## NITRIDE and CARBIDE BASED HT CERAMICS: Dr. Tarik Baykara - TUBITAK

- 8.00 - 8.30 Space Propulsion Research Program: Crossroads Among Combustion, Plasmas, And Materials  
**M. A. Birkan,\*** Air Force Office of Scientific Research - Bolling AFB - USA
- 8.30 - 9.00 Thermomechanical Properties of Refractory Hafnium and Zirconium Compounds  
**M. M. Opeka,\*** Naval Surface Warfare Center, MD - USA
- 9.00 - 9.30 High-Temperature Silicon Carbide Fibers  
**M. D. Sacks,\*** University of Florida, Gainesville, FL - USA
- 9.30 - 10.00 Creep of SiC/SiC Composites  
**R. E. Tressler,\*** K. L. Rugg, C. E. Bakis, Pennsylvania State Univ., University Park, PA - USA  
and J. Lamon - Laboratoire des Composites Thermostructuraux Domaine Universitaire-Pessac - France

## 10.00 - 10.30 COFFEE BREAK

## NITRIDE and CARBIDE BASED HT CERAMICS: Dr. Serene C. Farmer - NASA LeRC

- 10.30 - 11.00 Novel Developments of High-Temperature  $\text{Si}_3\text{N}_4$  Ceramics  
**M. Hoffmann,\*** Univ. of Karlsruhe, Karlsruhe - Germany
- 11.00 - 11.30 NNS of Monolithic and Composite HT Ceramics by HIP  
**H. T. Larker,\*** University of Lulea - Sweden
- 11.30 - 12.00 In-Situ Toughened Alpha SiALON  
**I-Wei Chen,\*** Mat. Sci. and Eng., Univ. of Pennsylvania, Philadelphia, PA - USA
- 12.00 - 12.20 New Developments in Alpha SiALON  
**H. Mandal,\*** Univ. Karlsruhe and Eskisehir Technical Univ. - Germany / Turkey
- 12.20 - 12.40 Titanium Carbide Composites for High Temperature Applications  
**N. Durlu,\*** TUBITAK, Gebze - Turkey
- 12.40 - 13.00 Synthesis of Fiber Reinforced Oxide Matrix Composites  
**R. Janssen,\*** Adv. Ceram. Group, Techn. Univ. Hamburg, Harburg - Germany

## 13.00 - 14.00 LUNCH

14.00 - Bus Departure to *Blue Mosque* and *Hagia Sophia*

18.30 - 19.00 Return to Conrad International Istanbul

**SPACEPROPULSIONRESEARCH  
PROGRAM: CROSSROADS  
AMONGCOMBUSTION,PLASMAS,  
ANDMATERIALS**

**Mitat A. Birkan\***

Air Force Office of Scientific Research  
110 Duncan Ave., Suite B115  
BOLLING AFB, DC 20332-0001  
USA

Basic research program in space propulsion aims to provide the fundamental knowledge required to improve and invent chemical and alternative non-chemical propulsion concepts that will increase performance and reliability, and/or decrease life-cycle and operational costs of Air Force space systems.

All dynamic systems operating at high temperatures require lightweight materials having high specific strength, with the goal of reducing the weight of spacecraft structures and propulsion systems. Low-cost processing technologies must also be pursued to reduce overall cost of space hardware. Engines operating in different regimes also require hydrogen, oxygen, corrosion resistant, and ultrahigh-temperature superalloys, ceramics, and polymer composites.

This seminar will include to give an overview of the operational regimes of chemical, electric, and beamed-energy launch and space propulsion systems, and their demands from the other disciplines, such as materials and structures, to overcome technological barriers.

**MECHANICAL,THERMAL,AND  
OXIDATIONPROPERTIES  
OFREFRACTORYHAFNIUMAND  
ZIRCONIUMCOMPOUNDS**

**M. M. Opeka,\*** I.G. Talmy, J.A. Zaykoski, E.  
J. Wuchina

Naval Surface Warfare Center, Carderock  
Division, West Bethesda, MD - USA  
S.J. Causey

Southern Research Institute, Birmingham, AL,  
USA

Fundamental properties (thermal conductivity, thermal expansion, Youngs Modulus, flexural strengths, and oxidation resistance) of hafnium and zirconium carbide, nitride, and diboride ceramics were investigated as a function of composition, including deviation of stoichiometry, SiC additions, and processing techniques. Flexure testing was conducted over a wide temperature range to determine the transition from elastic to plastic deformation.

For  $\text{HfC}_{0.98}$  and  $\text{HfB}_{1.95}$ , the transition temperatures differed significantly, being 2200 and 1100 °C, respectively. The transition temperature of HfC exhibited a strong stoichiometry dependence, decreasing from 2200 to 1100 °C for  $\text{HfC}_{0.67}$ . Thermal conductivities were measured for  $\text{HfC}_{0.98}$ ,  $\text{HfC}_{0.67}$ ,  $\text{HfN}_{0.92}$ , and  $\text{HfB}_{1.95}$  ceramics. The conductivity for  $\text{HfB}_{1.95}$  exceeded that for the other materials by a factor of 5 at room temperature and by a factor of 2.5 at 820 °C. Displacement reactions was used to synthesize  $\text{ZrB}_2$ -ZrC-SiC materials and their mechanical properties and oxidation behavior were evaluated.

## HIGH-TEMPERATURE SILICON CARBIDE FIBERS

Michael D. Sacks\*

University of Florida, Gainesville, FL 32611  
USA

Fine-diameter, SiC-based fibers with high strength and excellent thermomechanical stability were fabricated by dry spinning of organosilicon polymer solutions and subsequent heat treatment of the polymer fibers.

Fibers were prepared with both near-stoichiometric composition (i.e., ~67-70 wt% Si/~30-33 wt% C) and a composition with a substantial amount of excess carbon (i.e., overall composition of ~60 wt% Si/~40 wt% C). The fibers with near-stoichiometric composition were also prepared with boron nitride coatings. The effect of heat treatment temperature on fiber microstructure and mechanical properties was investigated. Fiber composition and microstructure were characterized using XRD, SEM, TEM, and elemental analysis techniques. Scanning Auger microprobe and X-ray photoelectron spectroscopy were used to characterize the surface chemistry of selected fibers. Fiber tensile strengths were determined after heat treatments at temperatures up to 1950 °C. Creep resistance was assessed from bend stress relaxation tests carried out using heat treatments at temperatures up to 1850 °C.

## CREEP OF SiC/SiC COMPOSITES

R. E. Tressler\* (1), K. L. Rugg (1\*), C. E. Bakis (2), and J. Lamon (3)

- 1 Department of Materials Science and Engineering
- 2 Department of Engineering Science and Mechanics  
The Pennsylvania State University  
University Park, PA 16802, USA
- 3 Laboratoire des Composites  
Thermostructuraux Domaine Universitaire  
3 allée de La Boétie, 33600 Pessac, France

Microcomposites of CVI SiC matrix/Hi-Nicalon fibers with a pyrocarbon interphase were crept at 1200-1400 °C and 140-450 MPa in an argon atmosphere. The creep behavior of the matrix and fiber constituents were studied separately using the same apparatus so that direct comparisons could be made using a simple rule of mixtures model for the uncracked composites. A series composite consisting of intact microcomposite, exposed fiber at the matrix crack, and the debonded region between these two regions was used to model the effects of matrix cracks. By performing unloading and loading hysteresis loops during the course of a creep run on cracked microcomposites, the requisite interfacial properties were derived for the model. Excellent fit to the experimentally observed creep curves were achieved with these models.

## NOVEL DEVELOPMENTS OF HIGH-TEMPERATURE $\text{Si}_3\text{N}_4$ CERAMICS

**Michael J. Hoffmann\***

University of Karlsruhe,  
Institute of Ceramics in Mechanical Engineering,  
Haid-und-Neu-Str. 7, D-76131 Karlsruhe,  
Germany

Silicon nitride ceramics are generally densified by liquid phase sintering using metal oxides as sintering additives. During cooling from processing temperature the additives form an amorphous or partially crystalline grain boundary phase. Creep resistant materials could only be obtained by a reduction of the additive content and by use of refractive additives. However, the additive minimization requires pressure assisted densification methods. The paper analyzes the potential of a Sinter-HIP process for complete densification of  $\text{Si}_3\text{N}_4$  with  $\text{Y}_2\text{O}_3$  contents varying between 2 and 10 wt. %. The process is divided in two steps: a pre-sintering step to achieve closed porosity and a pressure assisted step for the final densification. It will be shown that the required pore closure is strongly depend on the initial  $\text{Y}_2\text{O}_3/\text{SiO}_2$  ratio and the properties of the  $\text{Si}_3\text{N}_4$  starting powder, whereas the total additive content has only a minor influence. Furthermore, the influence of the applied pressure will be discussed. The materials reveal an excellent high-temperature strength and creep resistance with stationary creep rates below  $10^{-9} \text{ s}^{-1}$  at 100 MPa loading. However, by reducing the additive content the interfacial strength became stronger and the fracture mode changes from inter- to transgranular which gives a fracture toughness of only 2-3  $\text{MPam}^{1/2}$ . The obtained mechanical properties of the Sinter-HIPed samples were compared with conventionally HIPed ones prepared by glass encapsulation technique as well as sintered materials.

## NNS OF MONOLITHIC AND COMPOSITE HT CERAMICS BY HIP

**Hans T. Larker\***

University of Technology  
Praerstr. 4  
S 91531 Robertsfors  
Sweden

Improved properties of HT ceramics in general are achieved by compositional/processing R&D (compatible with sintering/shaping needs. Pressure is a great helper and HIP (hot isostatic pressing) offers flexibility.

Recent development within the EUREKA-AGATA car turbine project shows that improved HT material properties can be achieved, while at the same time fabricating components of complex shape, like turbine wheels, to near-net-shape (NNS). For such components a green body with close to homogeneous and isotropic characteristics are desired. A type of encapsulation, which creates no or minimal shear stresses at the surface of the green body during the shrinkage/sintering to full density, but at the same time prevents penetration into the body, will give optimal NNS results. Recent studies, e.g. by TEM, have confirmed that some encapsulation glass constituents can form new compounds with silicon nitride, at the very surface, which appear to help develop these desired characteristics.

Airfoil shapes can even be fabricated to net shape (NS) and with a novel interlayer at the surface were, (in the above EUREKA project), excellent tensile creep rupture results obtained, even with as-HIPed tensile bar surface, e.g. over 1500 h at 1350 °C and 75 MPa (partly) and 100 MPa with no failure. Non-homogeneous and non-isotropic ceramics, like fiber reinforced composites, may be fabricated using rigid, shape controlling tools on one or several sides. Particularly for large (and curved) panels can such use of a hot isostatic press be of advantage.

## DEVELOPMENT OF IN-SITU TOUGH ENDA LPHASIALON

I-Wei Chen\*

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University of Pennsylvania, Philadelphia, Pa  
19104-6272  
USA

Silicon nitride is currently used as cutting tools and structural components which demand high strength, toughness, hardness and corrosion resistance. Compared to composites and silicon carbide, it is relatively easy to manufacture to high density and consistent quality in large quantity. There are two forms of silicon nitride, alpha and beta form. The former is harder, but only the latter is currently used in engineering applications, because only this form can be given a microstructure resembling a whisker-reinforced composite, which gives it the necessary toughness and strength. We have recently discovered a method for the synthesis and processing of a tough alpha silicon nitride solid solution (SiAlON) with this kind of microstructure. The new material is 40% harder than beta silicon nitride and is more corrosion resistant. The processing science and the application tests of this material will be described in the presentation.

## NEW DEVELOPMENTS IN ALPHA SIALON CERAMICS

Hasan Mandal\*

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University, 26470, Eskisehir  
Turkey

Sialons are of interest for use as engineering materials for high temperature ( $>1000\text{ }^{\circ}\text{C}$ ) applications because they retain good mechanical properties at these temperatures, whereas competing metallic materials are weak and prone to corrosion. A characteristic disadvantage of all nitrogen ceramics is that an oxide additive is always included in the starting mix to promote densification, and this remains in the final product as a glassy phase distributed throughout the grain boundaries of the final microstructure. Since this glass melts at  $\sim 1000\text{ }^{\circ}\text{C}$ , the high temperature properties of the final ceramic are in fact determined by the properties of the grain-boundary glass.

The most common method of obtaining better high-temperature performance is to heat-treat the material at temperatures of  $1100\text{--}1350\text{ }^{\circ}\text{C}$  in order to devitrify the glass into a mixture of crystalline phases. More specifically it is desirable to convert the glass into a sialon phase plus only one other crystalline phase, the latter having a high melting point and also displaying a high eutectic temperature (max  $1400\text{ }^{\circ}\text{C}$ ) with the matrix sialon phase. Previous studies have shown that there are a limited number of possible metal-silicon-aluminium-oxygen-nitrogen compounds which satisfy these requirements. A better understanding of the phase relationships, stabilities of crystalline phases, microstructures and reaction chemistry in a variety of sialon systems has allowed improvement and optimization of high temperature performance in these systems.

In this respect, three new heat treatment techniques have been developed to improve and optimize the sialon ceramics which include:  
(1) a post-preparative vacuum heat treatment process to remove the grain boundary glass in a gaseous form,  
(2) above-eutectic heat treatment to crystallize new five component phases,  
(3) heat treatment of rare earth oxide densified sialon systems to tailor the microstructure and mechanical properties by reversible  $\alpha$ (alpha) -  $\beta$  sialon transformation.

# TITANIUM CARBIDE BASED COMPOSITES FOR HIGH TEMPERATURE APPLICATIONS

**Nuri Durlu\***

Materials Research Division, Marmara Research  
Centre,  
Tubitak, Gebze-Kocaeli  
Turkey

Titanium carbide based composites with nickel alloys and iron alloys as binder phase are currently used in high performance applications where wear and corrosion are the main sources of material failure. In order to extend the use of TiC-based composites, new binder systems should be developed. In this study our experimental work on TiC-based composites will be summarised with emphasis on new binder systems. The materials were prepared by powder metallurgy techniques. The sintering behaviour, the phase relations as determined by x-ray diffraction, and the mechanical properties (hardness and bending strength) of these composites will be presented.

# SYNTHESIS OF FIBER REINFORCED OXIDE MATRIX COMPOSITES

**R. Janssen,\* J. Wendorff, N. Claussen**  
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Advanced Ceramics Group, 21071 Hamburg  
Germany

Current research still addresses the need for fiber reinforced ceramic matrix composites (FRCMC) suitable for high temperature applications in oxidizing environments. Oxide matrix CMC's offer an encouraging route for materials capable of maintaining excellent stability in oxidizing atmospheres at high temperatures. The present paper deals with fiber reinforced alumina and mullite matrix ceramics fabricated by reaction bonding. In this process, metal-ceramic precursors containing reactive fine-grained Al particles are oxidized and subsequently sintered. Small fibers like polycrystalline Nextel 610 or amorphous Bayer SiBNC as well as large fibers like monocrystalline Saphikon EDF-fibers can be incorporated without any damage.

Beside synthesis of homogenous matrices, the fiber/matrix interaction has to be tailored in a respective way in order to get the desired damage tolerance. At present, we focus first on tailoring the elastic mismatch by adjusting the matrix porosity. This approach does not imply any fiber coatings and offers the potential for low-cost fabrication. However, for high application temperatures the matrix porosity has to be stabilized in order to avoid embrittlement of the composite during use. For alumina matrix composites, treatments in HCl containing atmospheres can be used to stabilize pores by vapor phase transport. Second, we look for new coatings offering the desired debonding behavior and advanced stability in oxygen at the same time also for dense composites. Beside ceramic coatings, also metal coatings like Platinum seems to be suitable. In the present paper, the different routes will be outlined by examples of recent research.

# FRIDAY

August 14, 1998

## OXIDE MATRIX COMPOSITES: Prof. Elizabeth Dickey - University of Kentucky

- 8.00 - 8.30 Interfaces in Oxide Composites  
**D. B. Marshall,\*** J. B. Davis, P. E. D. Morgan - Rockwell Science Center  
R. S. Hay - Air Force Research Lab. -USAF/AFRL/MLLN and A. Sayir - NASA
- 8.30 - 9.00 Layered-Structure Compounds for High Temperature Applications  
**W.T.Petuskey,\*** Dept. Chem. & Biochem., Arizona State U., Tempe, AZ-USA
- 9.00 - 9.30 Synthesis and Improvement of Sol-Gel-derived  $\text{LaAl}_{11}\text{O}_{18}$  Fiber Coatings  
**B. Saruhan,\*** German Aerospace Center, Koeln - Germany
- 9.30 - 10.00 Modeling of Non-Stoichiometry in Hexa - Aluminate Materials  
**A. N. Cormack,\*** Alfred Univ., Alfred, NY - USA

## 10.00 - 10.30 COFFEE BREAK

## HT OXIDE CERAMICS: Dr. Nuri Durlu - TUBITAK

- 10.30 - 11.00 Surface and Interface Properties of Alumina via Model studies of Microdesigned Interfaces  
**A. M. Glaeser,\*** et. al., UCLA, Berkeley, CA - USA
- 11.00 - 11.30 A Combined Experimental and Theoretical Approach to Atomic-Scale Structures in Complex Solids  
**S. Pennycook,\*** M. F. Chisholm, Y. Yan, K. Johnson, Oak Ridge Natl. Lab., Oak Ridge, TN - USA  
G. Duscher, N. D. Browning, Univ. of Illinois at Chicago, Chicago, IL - USA  
K. Johnson, V. P. Dravid, Northwestern University, Evanston, IL - USA  
S. T. Pantelides, Vanderbilt University, Nashville, TN - USA
- 11.30 - 12.00 Residual Stress Effects in Alumina-Zirconia Laminates  
**D. J. Green,\*** Peter Z. Cai, Penn State Univ., University Park, PA - USA  
V. M. Sglavo, Università di Trento, I-38050 Trento - ITALY
- 12.00 - 12.20 Residual Stresses in High-Temperature Ceramic Composites  
**E. Dickey,\*** University of Kentucky, Lexington, KY - USA
- 12.20 - 12.40 Rare Earth Doped High Temperature Ceramic Selective Emitters  
**D. L. Chubb,\*** NASA Lewis Research Center, Cleveland, OH - USA

## 12.40 - 13.30 LUNCH

## MULLITE BASED HT CERAMICS: Prof. Alan J. Ardell - UCLA

- 13.30 - 14.00 Suitability of Mullite for High Temperature Applications  
**R. Torrecillas,\*** INCAR - CSIC - Spain
- 14.00 - 14.30 Novel Alumina-Silicate Fiber/Mullite Matrix Composites with Favorable High Temperature Properties  
**B. Kanka and H. Schneider,\*** German Aerospace Center, Koeln - Germany



14.30 -15.00 High Temperature Single Crystal Properties of Mullite ( $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ )  
**W. M. Kriven,\*** and J. Palko, S. Sinogeikin and Jay D. Bass -Univ. of Illinois at Urbana-Champaign, Urbana, IL - USA  
A. Sayir - NASA Lewis Research Center, Cleveland, OH, USA  
G. Brunbauer, H. Boysen, F. Frey and J. Schneider -Ludwig Maximilian U., Germany

15.00 - 15.30 Phase Stability of Mullite/Alumina Composites  
D. M. Dabbs<sup>1,2</sup>, N. Yao,<sup>2</sup> and I. A. Aksay<sup>\*1,2</sup>  
1) Department of Chemical Engineering and  
2) Princeton Materials Institute  
Princeton University, Princeton, New Jersey 08544 - USA

15.30 - 16.00 Synthesis and Crystallization of YAG- And mullite-Composition Fibers  
**R. Weber,\*** B. Cho and P. Nordine - Containerless Research - Evanston, IL -USA

16.00 - 16.30 Creep and Microstructural Stability of Alumina - YAG Eutectics  
**L. E. Matson,\*** Air Force Research Lab. - USAF/AFRL/MLLN, OH - USA

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16.30 -18.00 **POSTERS:** Dr. A. Sayir - NASA Lewis Research Center

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Manufacture of Macro-Porous Ca-Hydroxyapatite Bioceramics  
**N. O. Engin,\*** and A. C. Tas, Metall. and Mat. Eng., Middle East Techn.Uni., Ankara - Turkey

Chemical Preparation of Stable Ca - Hydroxyapatite Powders At 37 °C In Body Fluids  
**D. Bayraktar\*** and A. C. Tas, Metall. and Mat. Eng., Middle East Techn.Uni., Ankara - Turkey

Modeling of Thermal Explosion Under Pressure in  $\text{B}_4\text{C}$  - Ti and BN-Ti Powder Blends  
M.Shapiro, V.Dudko, I.Gotman and **E.Y.Gutmanas\***, Technion, Haifa, 32000, Israel

Chemical Synthesis of Pure and Gd-Doped  $\text{CaZrO}_3$  Powders  
**I. E. Gönenli,\*** and A.C. Tas, Metall. and Mat. Eng., Middle East Techn.Univ., Ankara- Turkey

Toughening of Ceramic Composites by Transformation Weakening of Interphases  
**W. M. Kriven,\*** S. J. Lee, C. Huang, D. Zhu, Y. Xu and S. Mirek, Department of Materials Science and Engineering, University of Illinois at Urbana-Champaign, Urbana, IL 61801 - USA

Texture Development and Properties of Reaction-Based Alumina By Templated Grain Growth  
**E. Suvaci,\*** K. Watari, and G. L. Messing, Dep. Mat. Sci. & Eng., Penn State U., University Park, PA - USA

Processing of Steel/TiC Composites for Industrial Applications  
**N. Durlu,\*** Mat. Research Division, Marmara Research Centre, TUBITAK, Gebze - Turkey

Hard and Tough  $\alpha$ -Sialon Ceramics  
**H.Mandal\*<sup>1</sup>** and M.J.Hoffmann<sup>2</sup>, 1) Anadolu Uni., Ceramics Engineering, Eskisehir, 26470, Turkey  
2) Uni. of Karlsruhe, Institute for Ceramics in Mechanical Engineering, Germany

Capacitor-Discharge Joining of Ceramics  
**S. Turan\***, Anadolu Üniversitesi, Seramik Mühendisliği Bölümü, Eskisehir - Turkey

Wetting and Non-Wetting Behaviour of Interfaces In Ceramics  
**S. Turan\***, Anadolu Üniversitesi, Seramik Mühendisliği Bölümü, Eskisehir – Turkey

Microstructures of Mullite and SiC Whisker Reinforced Mullite  
**F. Kara\*** and Servet Turan, Anadolu Üniversitesi, Seramik Mühendisliği Bölümü, Eskisehir - Turkey

## INTERFACES IN OXIDE COMPOSITES

D. B. Marshall,\* J. B. Davis, P. E. D.

Morgan  
Rockwell Science Center

R. S. Hay  
WPAFB

A. Sayir  
NASA Lewis  
USA

Rare-earth phosphates form stable, weakly bonded interfaces with many oxides. They also appear to deform plastically relatively easily for such refractory materials. These properties make them potentially useful for use in oxide composites and in materials with special machining and wear properties. Studies of interface fracture and deformation in composites containing  $\text{LaPO}_4$  and various oxide fibers will be discussed.

## LAYERED-STRUCTURE COMPOUNDS FOR HIGH TEMPERATURE APPLICATIONS

W. T. Petuskey\*

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Arizona State University  
Tempe, Arizona, U.S.A. 85287-1604

The two dimensional nature of such compounds as layered perovskites, layered spinels, layered rutiles and layered silicates is reflected in their thermal and mechanical properties, even to very high temperatures. Some of these structures are of interest because of their easy cleaving capabilities and the ability to sustain considerable plastic damage through fracture basal fracture. They are of interest for high temperature use as weak interfacial phases in ceramic-matrix-composites. With the intention of identifying important structure and bonding characteristics, the thermal and mechanical properties of these materials are examined in terms of their crystal chemistry. The thermodynamic stability of each structure is also examined with an eye for designing optimal compositions and structures for high temperature applications.

# SYNTHESIS AND IMPROVEMENTS OF SOL-GEL-DERIVED $\text{LaAl}_{11}\text{O}_{18}$ FIBER COATINGS BY SEEDING

**Bilge Saruhan\***

German Aerospace Center-Institute for  
Materials Research, Cologne  
Germany

Lanthanum hexaluminate ( $\text{LaAl}_{11}\text{O}_{18}$ ) found application as an interface material in oxide-based fiber-reinforced composites, due to its unique structural formation and chemical compatibility with oxide matrices and fibers.

Sol-gel process is an effective way to coat fiber yarns with a thin (<500 nm) layer. A method for successful continuous coating of fibers has been developed. Promising compositions for fabrication of successful  $\text{LaAl}_{11}\text{O}_{18}$  coatings were established by systematical investigations of various sols and using alternative sol-gel-techniques such as seeding. All organic precursor results in the formation of  $\text{LaAlO}_3$ , eliminating the crystallization of  $\text{LaAlO}_3$ . Experiments show that addition of 2 wt.% phase pure  $\text{LaAl}_{11}\text{O}_{18}$ -seeds to the all-organic precursor combination lowers the crystallization temperature about 200°C and enables fiber coatings without fiber damage. Crystallization path by using various sol combinations was determined by means of DSC and XRD-measurements. The subsequent gel formation, drying and crystallization behaviors were investigated. The formation of  $\text{LaAlO}_3$  could be suppressed by changing the starting materials and processing route. Quality of  $\text{LaAl}_{11}\text{O}_{18}$  coatings on fibers depends strongly on the drying process. It is possible to obtain continuous coatings with no or little fiber bridging. Coating thickness which varies between 200-500 nm thickness have been achieved.

# MODELLING OF NON- STOICHIOMETRY IN HEXA- ALUMINATE MATERIALS

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Hexa-aluminate ceramics find a number of applications in high temperature environments, ranging from use as structural materials to use in catalytic combustors. Many of these materials are non-stoichiometric but, because of their multi-component nature, their defect chemistry is poorly understood.

The purpose of this presentation is to discuss the application of atomistic computer simulation methods to the determination of the point defect chemistry of hexa-aluminate and related systems. Such methods are particularly useful for these multi-component materials, because experimental methods are not always able to isolate the behaviour of individual defect species.

In one example, we show how non-stoichiometry in barium hexa-aluminate is accommodated, and in the second, we will use lanthanum hexa-aluminate to discuss how non-stoichiometry in poly-component systems may be predicted from the results of point defect energy calculations.

**SURFACE AND INTERFACE  
PROPERTIES OF ALUMINA VIA  
MODEL STUDIES OF MICRODESIGNED  
INTERFACES**

MIKITO KITAYAMA,<sup>1</sup> JAMES D. POWERS,<sup>2</sup>  
LAWRENCE KULINSKY<sup>3</sup> AND ANDREAS M. GLAESER<sup>3</sup>

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The development of methods for producing highly controlled internal voids in ceramics provided a new class of experimental methods for studying the high temperature properties of surfaces and interfaces in ceramics. Recent work, based on refined capabilities for producing microdesigned internal defect structures has broadened the range of problems that can be addressed. This paper reviews and previews recent research focussing on improving our understanding of surface diffusion in ceramics, providing experimentally determined values of surface energies in doped and undoped sapphire, and on developing new approaches to generating graded microstructures and single crystals by solid-state routes.

**ACOMBINED EXPERIMENTAL  
AND THEORETICAL APPROACH  
TO ATOMIC SCALE  
CHARACTERIZATION OF  
CERAMIC INTERFACES**

S. J. Pennycook<sup>1</sup>, M. F. Chisholm<sup>1</sup>, Y. Yan<sup>1</sup>,  
Duscher<sup>1,2</sup>, N. D. Browning<sup>2</sup>, K. Johnson<sup>3</sup>, V. I  
Dravid<sup>3</sup> and S. T. Pantelides<sup>1,4</sup>

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University, Nashville TN 37235*

The combination of Z-contrast imaging with electron energy loss spectroscopy (EELS) in a high-resolution STEM is a powerful method for the atomic level characterization of materials, giving direct information on atomic structure, impurity sites and valence. By avoiding the need for time-consuming searches of possible trial structures, such data also provides an ideal starting point for *ab initio* theoretical studies and a direct test of theoretical predictions. In MgO, Z-contrast revealed certain atomic columns to be significantly brighter than the bulk, and EELS established that the impurity is Ca. Theory then established that the presence of Ca impurities induces a structural transformation of the grain boundary. In a 36° Mn-doped grain boundary SrTiO<sub>3</sub>, atom column resolved EELS has shown significant changes in cation concentration and Mn valence at different atomic sites along the grain boundary. This synergistic approach of experiment and theory presents a viable means to determine the atomic nature of the defects responsible for the Schottky barriers in such electroceramic materials.

## RESIDUAL STRESSES IN ALUMINA-ZIRCONIA LAMINATES

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Vincenzo M. Sglavo  
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It has been established that a laminated structure can be utilized to toughen brittle materials, normally by an interfacial delamination process. It is, however, possible that other strengthening or toughening mechanisms may be available. In a recent study, it was shown that residual stresses can play an important role in both the processing and strength behavior of laminates. Symmetric alumina-zirconia laminates were formed by tape casting and pressureless sintering. During the firing process, residual stresses arise on heating because of the mismatch in sintering strain (-rate) and during cooling, because of thermal expansion mismatch. Both sources of stress were involved in the formation of transverse cracks during processing. In order to estimate the magnitude of these stresses, cyclic loading dilatometry was used to characterize the 'viscoelastic' behavior of the component layers during densification. These data were also used to predict the development of curvature in asymmetric laminates and this behavior was confirmed experimentally. In order to confirm the damage that occurs during densification, uniaxial stresses were applied to monolithic alumina during the firing process. Similar damage to that observed in the laminates was observed even with stresses below 1 MPa. The laminates were shown to have improved mechanical properties compared to the monolithic component materials but these results were primarily a result of the compressive residual stresses in the alumina layers.

## RESIDUAL STRESSES IN HIGH- TEMPERATURE CERAMIC COMPOSITES

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Department of Chemical and Materials  
Engineering, University of Kentucky  
USA

Residual stresses in composite materials arise from thermal expansion mismatches and interfacial constraints between the two phases and can have profound effects on the fracture behavior of the material. In fact, if the stresses become large enough, stress-relieving processes such as microcracking may occur at the interfaces in order to mitigate the stresses. While continuum models can be used to predict composite residual stresses, they must make implicit assumptions about the degree of constraint at the interface, because it is the interface which mediates the stress. It is therefore advantageous to experimentally measure residual stresses. Ultimately these measurements not only elucidate the stress state but, when compared to theory, give information about the degree of interfacial constraint. This seminar will present an experimental method for measuring residual stresses in directionally solidified ceramic eutectics, a class of materials which has much promise for high-temperature structural applications. Because directionally solidified eutectics are nearly single-crystal, special x-ray diffraction techniques have been developed to measure the residual strains. In the analysis the material is treated as fully anisotropic and the strain tensors, subsequently converted to stress tensors, are measured. It is discovered that enormous residual stresses can be present at room temperature in some as-processed eutectic materials (on the order of 1 GPa in NiO-ZrO<sub>2</sub>(c)). Plans for developing measurement techniques at elevated temperatures will be discussed.

## **RARE EARTH DOPED HIGH TEMPERATURE CERAMIC SELECTIVE EMITTERS**

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USA

Most materials at solid state densities emit in a continuous manner (blackbody or graybody emission). However, as a result of their atomic structure rare earth ions at solid state densities emit and absorb radiation in narrow wavelength bands. This selective emission property makes rare earth doped high temperature ceramic materials ideal emitter candidates for thermophotovoltaic (TPV) energy conversion systems. This paper will discuss the emissive properties of rare earth containing ceramics such as yttrium aluminum garnet (YAG). Both experimental and theoretical spectral emittance data will be presented.

## **SUITABILITY OF MULLITE FOR HIGH TEMPERATURE APPLICATIONS**

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INCAR CSIC  
La Corredoria s/n Ap. 73 Oviedo  
SPAIN

Mullite has never been considered to be suitable for high-strength applications at low temperatures because of its low fracture toughness value. Nevertheless, mullite stands out as a unique material for high temperature applications, not only as a single-phase material, but also as a matrix material in the development of high temperature composites. In order to design mullite structural components for high temperature applications stability and high temperature mechanical behaviour have to be studied.

The majority of high temperature mechanical studies of mullite have concentrated on measurement of the strength or creep deformation under conditions of four point bending or compression under static loads. These testing procedures are useful as an initial evaluation of failure strength or creep resistance but the complexity of stress system makes difficult the interpretation of the effect of material variables on the dominant creep mechanisms. Another approach uses fracture mechanics type test to study the growth of a major crack in the pre-cracked specimens. Because of experimental difficulty these tests have been carried out, up to now, at low temperatures and it is impossible to use results from these test to rationalise the crack growth mechanisms operative at high temperatures.

This work presents the results obtained during the last 5 years in a European collaborative research programme about high temperature mechanical behaviour of mullite based materials. An important part of this programme was related to the development of test methods for studies of tensile, flexural and compression creep up to 1400 °C, tension/compression cyclic fatigue life up to 1400 °C, crack growth rates under both cyclic and static loading at temperatures up to 1400°C using compact tension and double torsion specimens. Results obtained for different mullite based materials are discussed in terms of microstructural features, processing parameters and phase equilibrium diagrams.

# NOVEL ALUMINA-SILICATE FIBER/MULLITE MATRIX COMPOSITES WITH FAVORABLE HIGH TEMPERATURE PROPERTIES

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Germany

The use of ceramic composites as thermal protection shingles for combustion chambers and for other components exposed to hot gases (e.g. diffuser rings, liners) in aircraft turbine engines requires long-term (10,000h) mechanical stability and oxidation resistance up to 1,300 °C. Other important properties are damage tolerance, high thermal shock resistance, low thermal conductivity, chemical stability under long-term high temperature exposure, and low density. At present, continuous oxide fiber/mullite matrix composites are the materials which suit these conditions.

This report gives initial data on novel continuous alumino-silicate fiber (Nextel 720)/mullite matrix composites, fabricated through a low cost production route by winding slurry impregnated fibers on a cylindrical form. This tech requires no fiber / matrix interface. The composites undergo pressureless sintering in air at temperatures above 1300 °C for >15 min.

The material exhibits an excellent behavior at high temperature : The 3-point bending strength of the composite ranges between 200 and 300 MPa at room temperature and remains constant up to 1,200 °C does not decrease the strength at the material significantly. Also a constant high work of fracture (damage tolerance), caused by weak bonding between fibers and matrix is observed. The thermal shock resistance of the composites is excellent.

# HIGH TEMPERATURE SINGLE CRYSTAL PROPERTIES OF MULLITE ( $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ )

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University of Illinois at Urbana-Champaign,  
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A. Sayir,  
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G. Brunbauer, H. Boysen, F. Frey and J.  
Schneider, Institut für Kristallographie und  
Angewandte Mineralogie, Ludwig  
Maximilian Universität, München,  
Germany

Single crystal fibers of mullite were grown from the melt in air by a laser heated float zone method. Sound velocities and the single-crystal elastic moduli were measured by Brillouin spectroscopy from room temperature up to 1400 °C. This light scattering technique, which is based on the inelastic scattering of photons by acoustic phonons, does not require contact with the sample and is suitable for crystals of low symmetry.

Polycrystalline mullite made from hydrothermally grown stoichiometric ( $3\text{Al}_2\text{O}_3 \cdot 2\text{SiO}_2$ ) powders was also hot pressed at 1600 °C for 1hr in Ar atmosphere. The lattice parameters were measured by neutron diffraction as a function of temperature up to 1600 °C. X-ray diffractometry was also carried out up to 900°C and the data were evaluated by Rietveld analysis. The thermal expansion coefficients were determined.

## PHASE STABILITY OF MULLITE/ALUMINA COMPOSITES

D. M. Dabbs<sup>1,2</sup>, N. Yao,<sup>2</sup> and I. A. Aksay<sup>\*1,2</sup>

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2) Princeton Materials Institute

Princeton University, Princeton,

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USA

Differentiation of stable and metastable phase equilibria in the silica-alumina system has been the subject of numerous studies for more than a century. Among various unresolved issues, the differentiation of 2:1 mullite ( $2\text{Al}_2\text{O}_3 \bullet \text{SiO}_2$ ) from 3:2 mullite ( $3\text{Al}_2\text{O}_3 \bullet 2\text{SiO}_2$ ) is of importance especially in the development of mullite-matrix composites (e.g., with alumina). Contrary to the prevailing view that 3:2 and 2:1 mullites are simply the two extremes of a continuous solid range, we show that these two phases can co-exist as two distinct phases, providing proof for two solid solution ranges rather than a single one. In the presence of alumina, 3:2 mullite is the stable phase.

## SYNTHESIS AND CRYSTALLIZATION OF YAG- AND MULLITE-COMPOSITION FIBERS

R. Weber,\* Benjamin Cho and

Paul Nordine

Containerless Research, Inc.

906 University Place, Evanston, IL 60201,

USA

This talk will describe a new two-step process to manufacture inexpensive crystalline oxide fibers for high temperature structural applications. Strong and chemically homogeneous precursor fibers of 5-40  $\mu\text{m}$  diameter were synthesized at rates of up to 2 m/s by glass fiber pulling techniques from undercooled molten oxides. The precursor fibers were heat treated at temperatures from 900 - 1500 °C to make crystalline fibers with controlled grain size and properties. Tensile strengths of the precursor fibers were up to 6 GPa (900 ksi) for YAG- ( $\text{Y}_3\text{Al}_5\text{O}_{12}$ ) and mullite- ( $\text{Al}_6\text{Si}_2\text{O}_{13}$ ) compositions. Tensile strengths up to 3 GPa (450 ksi) have been obtained in the on-going crystallization research. Research to optimize fiber compositions and crystallization processes, and to scale up precursor fiber production will be presented and discussed.



# **MICROSTRUCTURAL STABILITY AND MECHANICAL PROPERTIES OF DIRECTIONALLY SOLIDIFIED ALUMINA/YAG EUTECTIC MONOFILAMENTS**

**L. E. Matson\***

Air Force Research Laboratory, Materials and  
Manufacturing Directorate,  
USAF/AFRL/MLLN

The size, shape, and distribution of two interpenetrating single crystal morphologies, such as those found in the Directionally Solidified (D.S.) Alumina /YAG Eutectic system (AYE), have a pronounced effect on microstructural stability, strength, toughness, and creep behavior. Microstructural scale, morphology, and crystallographic orientation relationships (O.R.) can be varied by manipulation of the D. S. process parameters as well as the size and shape of the fabricated part. The majority of the work reported upon here focuses on the properties 100  $\mu\text{m}$  monofilaments that contained the same O.R. throughout. However, the microstructure varied from a uniform "Chinese script" morphology to a duplex structure containing a colony with "rod/plate" morphology encased by a rim of "Chinese script".

Monofilaments have been produced with microstructure sizes less than 150 nanometers and strengths of 3 GPa. Similarly, the tensile creep resistance of aligned, fine-phased microstructures increases as the phase aspect ratio becomes very large (measured longitudinally with respect to the loading axis). This implies that the increase in the diffusional path length provides improved creep resistance. Monofilaments have been produced that show a steady-state creep rate of  $1 \times 10^{-8}$  /s. in air under a 375 MPa load at 1500 °C.

The inherent homogeneous coarsening in the AYE system is very slow below 1600°C and

obeys ( $t^{1/4}$ ) kinetics consistent with an interface boundary diffusion control mechanism. On the other hand, it has been observed that when the phase size is very fine (below 5  $\mu\text{m}$ ), anomalous heterogeneous coarsening can occur very rapidly at temperatures above 1400°C. This phenomenon is not a result of discontinuous coarsening, a mechanism usually observed in metallic eutectic systems, but is a result of a surface reaction between the YAG phase and silicon-containing contamination from the environment. This reaction allows the alumina phases to spheroidize rapidly in the presence of an yttrium-aluminum-silicate glass. The alternating alumina phases of the D. S. structure eventually impinge upon each other resulting in coarsening. These reaction defects limit the high temperature tensile strength and the rupture life of monofilaments. A clean environment, interface coatings, and the incorporation of the coated monofilaments into an alumina matrix should limit the occurrence of these reaction defects so that the superior mechanical properties of the system can be exploited.

The limitations of using the AYE monofilaments for CMCs will be its cost as well as monofilament weaving and lay-ups technologies. Only components requiring operations in air at ultra high temperatures for long periods will be practical. The scientific merit of being able to achieve high strength and creep resistance with fine interpenetrating aligned single crystal structures is significant and maybe far reaching. Ongoing work by other researchers in other eutectic systems as well as other processes which produce similar interpenetrating structures such as seeded bulk eutectics, rapidly solidified eutectic polycrystals, single crystal reinforcement preforming, controlled displacement reactions, and controlled decomposition of copolymers looks promising.

## POSTERS:

### MANUFACTURE OF MACRO-POROUS $\alpha$ -HYDROXYAPATITE BIOCERAMICS

**N. Ozgur Engin\*** and A. Cüneyt Tas  
Department of Metallurgical and Materials  
Engineering, Middle East Technical  
University, Ankara 06531, Turkey

Trabecular bones of almost all vertebrate organisms basically consist of macro-porous (65 to 70% interconnected porosity) bone mineral, i.e., calcium hydroxyapatite (HA:  $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ ). The macro-porosity observed in the trabecular bones then allows the ingrowth of the soft tissues and organic cells into the bone matrix.

Sub-micron, chemically uniform, and high phase-purity HA powders produced in our laboratory were mixed, under vigorous ultrasonification, with either methyl cellulose or polyethyleneimine of appropriate amounts in the form of an aqueous slurry of proper viscosity and thickness. The ceramic cakes produced in this way were then carefully dried in an oven in the temperature range of 70 to 90°C. The dried cakes of porous HA were then physically cut into various prismatic shapes. These parts were slowly heated in an air atmosphere to the maximum temperature of 1250°C.

The HA bioceramic parts obtained by this foaming technique were found to have tractable and controllable interconnected, open porosity in the range of 60 to 90%, with typical average pore sizes ranging from 100 to 250 microns. Sample characterization was mainly achieved by SEM (scanning electron microscope) studies.

### CHEMICAL PREPARATION OF STABLE $\alpha$ -HYDROXYAPATITE POWDERS AT 37°C IN BODY FLUIDS

**Defne Bayraktar\*** and A. Cüneyt Tas  
Department of Metallurgical and Materials  
Engineering, Middle East Technical  
University, Ankara 06531, Turkey.

Calcium hydroxyapatite (HA:  $\text{Ca}_{10}(\text{PO}_4)_6(\text{OH})_2$ ), which is heavily used in synthetic bone and dental applications, is the major inorganic phase of natural human and animal hard tissues. Calcium hydroxyapatite has been synthesized in this work as high phase-purity and as extremely fine (with an average particle size of 50 nm) bioceramic powders from the starting materials of calcium nitrate tetra-hydrate and di-ammonium hydrogen phosphate. "Synthetic body fluid" solutions of a novel composition, prepared in our laboratories, have been used instead of distilled or de-ionized water during the chemical precipitation runs.

The chemical precipitation experiments were carried out at the constant temperature of 37°C and at a constant solution pH of 7.4. The "biomimetic" HA powders thus synthesized displayed superior thermal decomposition stability when heated in air. These powders were found not to decompose into the Whitlockite ( $\beta$ -TCP:  $\text{Ca}_3(\text{PO}_4)_2$ ) phase even after heating for 6 hours in a stagnant dry air atmosphere at 1600°C.

The chemical and structural characterization of SBF-synthesized HA powders were performed by ICP-AES (inductively-coupled plasma – atomic emission spectroscopy), powder XRD (X-ray diffraction), SEM (scanning electron microscopy), EDXS (energy-dispersive X-ray spectroscopy), FT-IR (Fourier-transformed infrared spectroscopy) and Rietveld analysis.

## **POSTERS:**

### **MODELING OF THERMAL EXPLOSION UNDER PRESSURE IN $B_4C$ - Ti AND BN-Ti POWDER BLENDS**

M. Shapiro, V. Dudko, I. Gotman and  
**E.Y. Gutmanas\***  
Technion, Haifa, 32000,  
Israel

The process of reactive *in situ* synthesis of dense  $TiB_2/TiC$  and  $TiB_2/TiN$  ceramic matrix composites from  $B_4C$ -Ti and BN-Ti powder blends with and without additions of Ni has been modeled. The objective of modeling was the determination of optimal thermal conditions preferable for production of fully dense ceramic matrix composites. Towards this goal, heat transfer and combustion in dense and porous ceramic-metal blends have been investigated during monotonous heating at a constant rate. The process has been modeled using a heat transfer-combustion model with kinetic parameters determined from the differential thermal analysis of the experimental data. The kinetic burning parameters and the model developed have been further used to describe the process of combustion synthesis in a restrained die under pressure. It has been shown that the heat removal from the reaction zone affects the ignition temperature of thermal explosion.

### **CHEMICAL SYNTHESIS OF PURE AND Gd-DOPED $CaZrO_3$ POWDERS**

**I. Erkin Gönenli\*** and A. Cüneyt Tas  
Department of Metallurgical and Materials  
Engineering, Middle East Technical  
University, Ankara 06531,  
Turkey

Aqueous solutions of calcium chloride ( $CaCl_2$ ) and zirconium oxychloride ( $ZrOCl_2 \cdot 8H_2O$ ), in appropriate volumetric amounts, were used as the starting materials in the synthesis of phase-pure  $CaZrO_3$  powders. Rare earth element dopings (in the range of 5 to 25 at%) were performed by using the aqueous chloride solutions of Gadolinium. Preparation of the desired powders were achieved either by self-propagating combustion synthesis or by the technique of acid-base titration in the presence of Ca-EDTA.

Crystal chemical aspects of the resulting powders were studied by Rietveld analysis, as a function of the amounts of the dopant incorporated into the crystal structures and the powder calcination temperature (1200 to 1400°C), and then compared with those of pure  $CaZrO_3$ .

Morphological properties of the synthesized powders were investigated by the SEM (scanning electron microscopy) studies. Qualitative chemical analyses of the samples were performed by EDXS (energy-dispersive X-ray spectroscopy).

## **POSTERS:**

### **TOUGHENING OF CERAMIC COMPOSITES BY TRANSFORMATION WEAKENING OF INTERPHASES**

**W. M. Kriven,\* S. J. Lee, C. M. Huang, D.  
Zhu, Y. Xu and S. M. Mirek**

Department of Materials Science and  
Engineering  
University of Illinois at Urbana-Champaign  
Urbana, IL 61801,  
USA

A new concept for debonding of interphases between fibers and matrix or between laminates in oxide ceramic composites is introduced. It is based on a thermal or shear stress induced phase transformation which is accompanied by a volume contraction or significant shape change, leading to microcracks. Thermally induced transformation above the critical particle size was demonstrated in enstatite ( $\text{MgO} \cdot \text{SiO}_2$ ) due to an orthorhombic protoenstatite (PE) to monoclinic clinoenstatite (CE) transformation which is accompanied by a 5.5% volume contraction during cooling at 865 °C, forming intragranular microcracks. Thermal or shear stress induced transformation was also observed in (Ca, Al)-doped cristobalite ( $\text{SiO}_2$ ) where the cubic (b) to tetragonal (a) transformation occurs on cooling at 265 °C, with a ~3.2 % volume contraction. Laminates of mullite/cordierite separated by cristobalite interphases were fabricated to optimize thermal expansion mismatch. Crack propagation was observed within the transformed interphases and graceful failure was observed in 4-point flexure testing.

### **TEXTURE DEVELOPMENT AND PROPERTIES OF REACTION-BASED ALUMINA BY TEMPLATED GRAIN GROWTH**

**E. Suvaci\*, K. Watari, and G. L. Messing,**  
Department of Materials Science and  
Engineering, Materials Research Laboratory,  
The Pennsylvania State University,  
University Park, PA 16802,  
USA

Highly textured (70-80 vol.%), dense (~95% TD) alumina ceramics were successfully fabricated by a new processing route which uses a mixture of Al metal powder, alumina powder, alumina platelet (template) particles and a liquid phase former. The process involves the use of dry forming techniques (e.g., uniaxial pressing, and roll compaction) to align plate-like template particles. This study demonstrates the effect of processing on texture and properties of reaction bonded alumina. The reaction-based processing was analyzed by thermogravimetric analysis. The microstructure evolution and texture development were characterized by scanning electron microscopy and X-ray diffraction. Fracture toughness was measured by indentation technique, and laser flash method was utilized to measure thermal conductivity of the textured ceramics as a function of direction.

## POSTERS:

### PROCESSING OF STEEL/TiC COMPOSITES FOR INDUSTRIAL APPLICATIONS

**Nuri Durlu\***

Materials Research Division, Marmara  
Research Centre,  
Tubitak, Gebze-Kocaeli,  
Turkey

The major factors which influence the tool life in manufacturing operations are wear resistance, toughness, and hardness at high temperatures. If appropriately designed, these property requirements can be met successfully by steel/TiC composites. Wire drawing dies, stamping dies, punches and guide rolls are some of the tools which can be made from steel/TiC composites. This study deals with the near net shape processing of several steel/TiC composite proto-types. Powder metallurgy techniques were utilised to develop steel/TiC composites which contain less than 50vol%TiC, and hardness values above 60 Rc .

### HARD and TOUGH $\alpha$ -SiAlON CERAMICS

**H.Mandal\*, M.J.Hoffmann\*\***

\*Anadolu University, Department of  
Ceramics Engineering, Eskisehir, 26470,  
Turkey

\*\*University of Karlsruhe, Institute for  
Ceramics in Mechanical Engineering,  
Germany

Yb, Nd/Ca and Ce/Ca containing  $\alpha$ -sialon starting compositions have been densified by either pressureless sintering or capsule free sinter/HIP'ing using "conventional"  $\alpha$ -Si<sub>3</sub>N<sub>4</sub> powder. X-ray diffraction studies of both compositions after sintering revealed that only  $\alpha$ -sialon was observed as a matrix phase. Microstructural characterization of the relatively high nitrogen rich starting composition resulted in a typical equiaxed grain morphology, as expected. But the relatively less nitrogen rich starting composition exhibited surprisingly an elongated grain morphology with typical grain length of 5-20  $\mu$ m and aspect ratios of 4-15. The mechanical property measurements on the material with equiaxed grains showed a hardness value of 19.76 GPa and a toughness value of 3.96 MPam<sup>1/2</sup> as expected. The  $\alpha$ -sialon material with elongated grains proved to have a hardness value of 19.30 GPa (HV10) but fracture toughness value of 6.31 MPam<sup>1/2</sup>. Crack deflection, crack bridging and grain pull-out mechanisms were accounted for the significant improvement in fracture toughness.

## **POSTERS:**

### **CAPACITOR-DISCHARGE JOINING OF CERAMICS**

**Servet Turan\***

Anadolu Üniversitesi, Mühendislik-Mimarlık  
Fakültesi, Seramik Mühendisliği Bölümü,  
Yunussemre Kampüsü, 26470 Eskisehir,  
Turkey

A capacitor-discharge joining technique has been used to fabricate alumina-metal-alumina, zirconia-metal-zirconia, silicon carbide-metal-silicon carbide and silicon nitride-metal-silicon nitride joints using thin foils of aluminium, titanium or amorphous alloys as interlayers. The technique involves passing a high energy pulse through a conductive interlayers, so converting the interlayer into a liquid-vapour "plasma" which wets and bonds the pieces of ceramic being joined.

In order to understand the bonding mechanism, the interfacial microstructures of the bonds were examined in great detail using an optical microscope, a scanning electron microscope, conventional as well as high resolution transmission electron microscopes and a dedicated scanning transmission electron microscope. The bond strengths of the joints were assessed by shear testing.

The results obtained from the microstructural investigations coupled with the shear test measurements revealed that the highest bond strengths were obtained when there was a distinct reaction layer between the ceramic substrate grains and the interlayer. In this presentation, a new ceramic joining technique will be introduced and the relationship between microstructure and joint strength will be demonstrated.

### **WETTING AND NON-WETTING BEHAVIOUR OF INTERFACES IN CERAMICS**

**Servet Turan\***

Anadolu Üniversitesi, Mühendislik-Mimarlık  
Fakültesi, Seramik Mühendisliği Bölümü,  
Yunussemre Kampüsü, 26470 Eskisehir,  
Turkey

Extensive studies on silicon nitride-silicon carbide particulate composites containing hexagonal boron nitride have shown clear evidence that some interfaces are wetted with intergranular films whereas some of them were free from such films. When intergranular films observed, its thickness found to be constant when the crystals at either side of the film is isotropic, whereas if the crystals are highly anisotropic then the film thickness varied depending on the misorientation across the interface in contrast to the previous observations and theoretical expectations.<sup>1</sup> In addition, while boron nitride-silicon carbide interphase boundaries contained intergranular films, in the same type of composites boron nitride-silicon nitride interphase boundaries did not. The reasons for this difference in behaviour from that predicted by Clarke's model<sup>1</sup> will be discussed.

1. D.R. Clarke *J. Am. Ceram. Soc.*, 1987, 70, 15-22.

# MICROSTRUCTURES OF MULLITE AND SiC WHISKER REINFORCED MULLITE

**Ferhat Kara\*** and Servet TURAN

Anadolu Üniversitesi, Mühendislik-Mimarlık Fakültesi, Seramik Mühendisliği Bölümü, Yunus Emre  
Kampüsü, 26470 Eskişehir,  
Turkey

Microstructures of pure mullite produced from aluminium sulphate-colloidal silica and boehmite-colloidal silica mixtures by reaction sintering and of SiC whisker reinforced-mullite composites produced by hot isostatic pressing of 25% SiC whisker-AlOOH colloidal silica mixtures were examined by a number of transmission electron microscopy based techniques including diffuse dark field, fresnel fringe defocus and high resolution electron microscopy.

It was observed that pure mullite ceramics free from glassy phase at triple junctions and grain boundaries could be produced from these mixtures. If glass occurs in mullite due to incomplete reaction between alumina and silica components or due to release of silica from mullite structure, wetting of grain boundaries found to be depend on the microstructural development, e.g mainly non-wetted grain boundaries were observed in mullite experiencing extensive grain growth.

In SiC whisker reinforced mullite composite, interfaces between mullite and SiC contained amorphous phases. Near the interface, mullite lattice was heavily deformed due to high pressure (200 MPa) applied during hot isostatic pressing.

**SATURDAY**

**August 15, 1998**

**THERMOMECHANICAL STABILITY:** Prof. Cuneys Tas - Middle East Tech U. -  
Ankara

8.00 - 8.30 Mechanisms of Deformation of Structural Ceramics at High Temperatures  
**S. M. Wiederhorn,\*** Nat. Inst. of Standards and Tech., Gaithersburg, MD - USA

8.30 - 9.00 Microstructural Stability at Elevated Temperatures  
**A. J. Ardell,\*** Dept. of Mat. Sci. and Eng. Uni. of California, LA, CA - USA

9.00 - 9.30 Phonon Excitations and Related Thermal and Mechanical Properties of  $\text{Si}_3\text{N}_4$ , AlN  
and GaN Ceramics: A Neutron - Scattering Study  
**C.-K. Loong,\*** Argonne Nat. Lab., Argonne, Illinois - U. S. A.

9.30 - 10.00 Multimillion Atom Molecular Dynamics Simulations Of High Temperature  
Ceramics On Massively Parallel Computers  
**R. K. Kalia,\*** A. Nakano, A. Omeltchenko, K. Tsuruta, and P. Vashishta  
Dep. of Phy. and Astronomy, Louisiana S. U., Baton Rouge, LA - USA

**10.00 - 10.30 COFFEE BREAK**

**THERMOMECHANICAL STABILITY:** C.-K. Loong,\* Argonne Nat. Lab., Illinois - U. S. A

10.30 - 11.00 Atomic Stresses in  $\text{Si/Si}_3\text{N}_4$  Nanopixel - 10 Million Atom Molecular Dynamics  
Simulation on Parallel Computers  
**P. Vashishta,\*** Dep. of Phy. and Astronomy, Louisiana S.U., Baton Rouge, LA -USA

11.00 - 11.30 Anisotropic Properties in Hot Pressed Silicon Nitride - Silicon Carbide Platelet  
Reinforced Composites  
M. Poorteman, **P. Descamps\*** and F. Cambier, Belgian Ceramic Res. C., Mons -  
A. Poulet and J.C. Descamps - Mat. Sci., Polytechnic Faculty of Mons - Belgium

11.30 - 12.00 Use of Valence Electron Number to Identify New Compounds with High  
Reflectance and/or Refractoriness  
**M. M. Opeka,\*** and J. A. Zaykoski, Naval Surface Warfare Center, MD - USA

12.00 - 12.20 Chemical Preparation of HT Oxide Powders  
**A. C. Tas,\*** Metall. and Mat. Eng., Middle East Techn.Uni., Ankara - Turkey

12.20 - 12.40 Interfaces in Yttrium - Doped  $\alpha\text{-Al}_2\text{O}_3$   
**M. A. Gülgün,\*** V. Putlayev, and M. Rühle, Max-Planck Ins., Stuttgart-GERMANY

12.40 - 13.00 Directionally Solidified Eutectic Ceramics  
**A. Sayir,\*** and S. C. Farmer, NASA LeRC, Cleveland, OH - USA

**13.00- 14.00 LUNCH**

14.00 - **BUS DEPARTS TO DOCK**

14.30 -18.00 **BOSPHORUS TOUR WITH BOAT (TASKENT)**

18.00 - 21.00 **BANQUET DINNER AT "AQUARIUS RESTORAN" SARIYER**  
*(near the Black Sea)*

22.30 **RETURN TO CONRAD INTERNATIONAL ISTANBUL BY BUS**



# MECHANISMS OF DEFORMATION OF STRUCTURAL CERAMICS AT HIGH TEMPERATURES

Sheldon M. Wiederhorn\*  
National Institute of Standards and  
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Gaithersburg, MD 20899  
USA

Current grades of ceramic materials have two types of microstructures. In one, the grains are separated by relatively pure grain boundaries and deformation occurs by classical diffusional processes. In the other, liquid phase sintering is used to process the material and the grain boundaries are covered by residual sintering aid. In this paper we discuss both types of materials. The first class of structural material can be typified by silicon carbide, which shows excellent creep resistance at temperatures over 1500 °C. Depending on the technique of manufacture, a substantial variation in the creep resistance of this material has been observed. Sintering aids that enhance the diffusivity of the silicon carbide, substantially reduce the creep resistance of the material. Far higher creep resistance is obtained by non-classical techniques of processing. The principal drawback of the single phase materials is their low toughness, which ranges from about 2 - 4 MPa•m<sup>1/2</sup> for silicon carbide. Materials with a second phase at the grain boundaries have a higher toughness, 6 - 10 MPa•m<sup>1/2</sup> for silicon nitride, but a substantially lower creep resistance. Deformation of these materials at high temperatures depends the refractoriness of the sintering aid, and occurs by cavity formation at multigrain junctions. A comparison of the two types of materials is made and methods of improving creep resistance are suggested.

# MICROSTRUCTURAL STABILITY AT ELEVATED TEMPERATURES

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Engineering  
University of California  
Los Angeles, CA 90095-1595  
USA

The stability of dispersed phases at elevated temperatures will be reviewed in this presentation. The microstructures considered span the gamut encountered in high-temperature materials. This includes precipitates in Ni-base superalloys, which coarsen by diffusion-controlled Ostwald ripening, and rods, fibers or lamellae, which provide most of the elevated-temperature strength of directionally solidified metallic or ceramic eutectics. The coarsening of eutectic microstructures can occur by several different processes, with rod eutectics coarsening by either two-dimensional Ostwald ripening, or by the movement of microstructural faults, each consisting of a termination and a branch. Lamellar eutectics coarsen either by the migration of similar faults or by discontinuous coarsening, which is another, totally different mechanism. In this paper all the mechanisms of microstructural coarsening will be reviewed and examples of the different types of behavior will be presented, with examples from ceramic systems wherever possible.

**PHONON EXCITATIONS AND  
RELATED THERMAL AND  
MECHANICAL PROPERTIES OF  
Si<sub>3</sub>N<sub>4</sub>, AlN AND GaN CERAMICS: A  
NEUTRON-SCATTERING STUDY**

**C.-K. Loong\***

Argonne National Laboratory, Argonne,  
Illinois 60439-4814,  
U. S. A.

Structural components and semiconductor devices based on silicon nitride, aluminum nitride and gallium nitride are expected to function more reliably at elevated temperatures and at higher levels of performance because of the strong atomic bonding in these materials. The degree of covalency, lattice specific heat and thermal conductivity are important design factors for the realization of advanced applications. We have determined the crystal structures and phonon densities of states of these ceramics by the method of neutron scattering. The results provide a microscopic interpretation of the mechanical and thermal properties. Moreover, experimental data of the static structures and dynamic excitations of atoms are essential to the validation of interparticle potentials employed for molecular-dynamics simulations of high-temperature properties of multi-component ceramic systems. We present an overview of neutron-scattering investigations of the crystal phases and phonon excitations, and calculations of the phonon dispersion relations and thermodynamic properties of Si<sub>3</sub>N<sub>4</sub>, AlN and GaN. The results are compared with those of the corresponding oxides such as SiO<sub>2</sub> and Al<sub>2</sub>O<sub>3</sub>. Work performed at Argonne National Laboratory is supported by the U. S. DOE-BES under the contract No. W-31-109-ENG-38.

**MULTIMILLION ATOM  
MOLECULAR DYNAMICS  
SIMULATIONS OF HIGH  
TEMPERATURE CERAMICS ON  
MASSIVELY PARALLEL  
COMPUTERS**

**Rajiv K. Kalia,\*** Aiichiro Nakano, Andrey  
Omeltchenko,  
Kenji Tsuruta, and Priya Vashishta

Concurrent Computing Laboratory for  
Materials Simulations Department of Physics  
and Astronomy Department of Computer  
Science Louisiana State University, Baton  
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USA

Large scale molecular dynamics simulations are performed to investigate the properties of silicon carbide, silicon nitride and diamond coatings. The simulations are performed with highly efficient, portable and scalable, multiresolution algorithms on massively parallel machines. Results for structural correlations, mechanical behavior, and crack propagation in silicon nitride and silicon carbide and hypervelocity impact damage in diamond coatings will be presented.

# ATOMIC STRESSES IN Si/Si<sub>3</sub>N<sub>4</sub> NANOPIXEL - 10 MILLION ATOM MOLECULAR DYNAMICS SIMULATION ON PARALLEL COMPUTERS

P. Vashishta,\*

Concurrent Computing Laboratory for Materials  
Simulations, Department of Physics &  
Astronomy, and Department of Computer  
Science, Louisiana State University, Baton  
Rouge, Louisiana 70803-4001 - USA

Silicon nitride and silicon carbide are among the two most important high temperature materials. Silicon carbide is used as a device material (electronic ceramic) in high temperature electronic devices and silicon nitride and silica deposited on silicon substrate are used as dielectric layers in semiconductor technology. Experimental studies indicate that Si/Si<sub>3</sub>N<sub>4</sub> interface offers significant advantages over Si/SiO<sub>2</sub>, such as higher density, higher dielectric constant, and better radiation hardness. Si<sub>3</sub>N<sub>4</sub> on Si films are grown by plasma-enhanced chemical vapor deposition (PECVD) followed by annealing processes. These films are usually amorphous, but coherent interface between crystalline Si and Si<sub>3</sub>N<sub>4</sub> has also been observed.

We have combined first-principle calculations of charge transfer at the interface with the interaction potential models for bulk Si and Si<sub>3</sub>N<sub>4</sub> to produce a model for the Si/Si<sub>3</sub>N<sub>4</sub> interface. Using these interatomic potentials, million atom molecular dynamics simulations have been performed to characterize the structure of Si(111)/Si<sub>3</sub>N<sub>4</sub>(0001) and the Si(111)/ $\alpha$ -Si<sub>3</sub>N<sub>4</sub> interfaces. Mechanical stability of the interface have been investigated by looking at the crack propagation and fracture in the system under an applied strain.

Ten million atom multiresolution molecular-dynamics simulations are performed on parallel computers to determine atomic-level stress distributions in a 54 nm nanopixel on a 0.1 micron silicon substrate. Effects of surfaces, edges, and lattice mismatch at the Si(111)/Si<sub>3</sub>N<sub>4</sub>(0001) interface on the stress distributions are investigated. Stresses are found to be highly inhomogeneous in the nanopixel. The top surface of silicon nitride has a compressive stress of +3GPa and the stress is tensile, -1GPa, in silicon below the interface. These simulation methods can also be applied to other semiconductor/ceramic interfaces as well as to metal/ceramic and ceramic/ceramic interfaces.

# ANISOTROPIC PROPERTIES IN HOT PRESSED SILICON NITRIDE - SILICON CARBIDE PLATELET REINFORCED COMPOSITES

M. Poorteman<sup>1</sup>, P. Descamps<sup>1\*</sup> and F.  
Cambier<sup>1</sup>

A. Poulet<sup>2</sup> and J.C. Descamps<sup>2</sup>

<sup>1</sup> Belgian Ceramic Research  
Centre, Mons - Belgium

<sup>2</sup> Materials Science Department,  
Polytechnic Faculty of Mons - Belgium

During the fabrication of ceramic composites by uniaxial hot pressing, grain orientation with respect to the pressing direction can occur. As a consequence, the mechanical properties (toughness for instance) can become direction dependent. Indeed, when the reinforcement is obtained by crack deflection or crack bridging mechanisms, the contribution to the toughness will depend on the orientation of the interacting grain with respect to the crack plane. Moreover, the interaction of the crack with the reinforcing particle will also depend on the grain morphology. For instance, the orientation dependency of the contribution to reinforcement of one-dimensional fibrelike grains can be quite different compared to two-dimensional platelets.

In this work, we will discuss the anisotropic properties of hot pressed platelet reinforced silicon nitride composite compared to the monolithic material. In those materials, reinforcement can mainly be attributed to reinforcing mechanisms such as crack deflection and crack bridging by grains with high aspect ratio (platelets or silicon nitride). The anisotropy of the mechanical properties of the monolithic material and the composite will be assessed by SEM and TEM observation as well as by X-Ray texturing. Taking into account those analyses, the mechanical properties variation will be discussed in terms of grain orientation and grain morphology.

# USE OF VALENCE ELECTRON NUMBER TO IDENTIFY NEW COMPOUNDS WITH HIGH REFLECTANCE, AND/OR REFRACTORINESS

M. M. Opeka,\* J.A. Zaykoski  
Naval Surface Warfare Center, Carderock  
Division, West Bethesda, MD  
USA

Melting point and reflectance were correlated to the number of valence electrons for compounds with NaCl-, AlB<sub>2</sub>-, CaB<sub>6</sub>-, CsCl-, and CaTiO<sub>3</sub>-type crystal structures. Maximum melting points for these compounds are observed at a particular valence electron number. For the AlB<sub>2</sub>-type structure this number is 10 e<sup>-</sup>/formula unit. Based on this number, the (Ta<sub>0.5</sub>Sc<sub>0.5</sub>)B<sub>2</sub> and ScBSi compositions were predicted to form stable, ordered ternary compounds. Materials in these two systems were prepared. Their phase composition and properties are under evaluation and will be discussed. For reflectance behavior, it was hypothesized that additional valence electrons above that required for maximum structure stability would result in higher reflectance in the visible to near infrared wavelength range. For compounds of all five previously mentioned structure types, this hypothesis was confirmed based on published data and experimental results.

# CHEMICAL PREPARATION OF HT OXIDE POWDERS

A. Cuneyt Tas\*

Department of Metallurgical and Materials  
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A number of aluminates, zirconates, titanates, borates, silicates and phosphates have been prepared by chemical means from aqueous solutions in the form of chemically homogeneous, sub-micron ceramic powders.

Lanthanum monoaluminate (LaAlO<sub>3</sub>) powders were synthesized either by self-propagating combustion synthesis (SPCS) or homogeneous precipitation from the nitrate solutions of the respective cations. Sub-micron powders of all the binary compounds of the CaO-Al<sub>2</sub>O<sub>3</sub> system have been prepared by the SPCS technique. Single phase and Gd-doped (5 to 25 at%) CaZrO<sub>3</sub> powders were synthesized by a novel acid-base titration technique in the presence of a chelating agent (i.e., Na-EDTA). Pure or Dy-doped (0.8 at%) BaTiO<sub>3</sub> powders have been prepared at 90 °C by a modified hydrothermal synthesis technique in an air atmosphere. Dy-doping in these powders was shown to inhibit the anomalous grain growth during the sintering of BaTiO<sub>3</sub>. PZT powders were synthesized by homogeneous precipitation and the PZT precursors were found to crystallize into single phase sub-micron PZT after calcination in an air atmosphere at 650 °C for 6 hours.

Short and randomly-oriented whiskers of aluminum borate (9Al<sub>2</sub>O<sub>3</sub>•2B<sub>2</sub>O<sub>3</sub>) were synthesized by a flux-growth method. A new XRD pattern was derived from our experimental data. Sub-micron, monodisperse powders of Enstatite, Cordierite and Mullite were synthesized from TEOS and nitrate solutions, without using any organic agents. Mullite powders were also prepared by homogeneous precipitation in the presence of urea in the mother liquors. Calcium phosphate powders (HA, TCP, or bi-phasic composites of HA-TCP) were prepared in either de-ionized water (55 °C) or synthetic body fluid solutions (37°C). Biomimetic coating of carbonated and amorphous HA powders on ceramic, metal or polymer substrates were shown to take place when these materials were immersed in body fluid solutions kept at 37 °C and pH = 7.4. Porous (average pore size of 250 μm, with a relative porosity in the range of 50 to 85%) HA bioceramics were formed by a novel foaming technique.

## INTERFACES IN YTTRIUM-DOPED $\alpha$ - $\text{Al}_2\text{O}_3$

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Yttrium-doped  $\alpha$ - $\text{Al}_2\text{O}_3$  has a creep rate that is approximately two orders of magnitude lower than the creep rate of pure alumina. This remarkable improvement in creep is closely related to the limited solubility of yttrium in bulk alumina (10 ppm) and its consequent segregation/precipitation behavior. A comprehensive electron microscopy investigation was undertaken to study the microstructure, microchemistry and crystallography of interfaces in a 1000 at. ppm (Y/Al) Y-doped  $\alpha$ - $\text{Al}_2\text{O}_3$ . It is aimed to establish the link between the segregation/precipitation behavior, microstructure, and the improved creep properties of the material. At this dopant level, yttrium does not only segregate to grain boundaries (GB), but also precipitate out as YAG crystals ( $\text{Y}_3\text{Al}_5\text{O}_{12}$ ). Various interfaces (homo and heterophase boundaries) were studied with conventional TEM for their crystallography and their frequency in the microstructure. Energy dispersive spectrometry (EDS) attached to a dedicated STEM was used to quantitatively characterize the microchemistry of different types of boundaries. Yttrium level at GBs was observed to vary significantly (0 atoms/ $\text{nm}^2$  to 3.2 atoms/ $\text{nm}^2$ ) depending on the type of the boundary. A variation of the segregant concentration within the same boundary indicated a non-equilibrium segregation of the dopant. An extensive electron loss near edge structure (ELNES) investigation of yttrium-doped grain boundaries and yttrium aluminate compounds ( $\text{Y}_2\text{O}_3$ ,  $\text{Y}_4\text{Al}_2\text{O}_9$ ,  $\text{YAlO}_3$ ,  $\text{Y}_3\text{Al}_5\text{O}_{12}$  and  $\alpha$ - $\text{Al}_2\text{O}_3$ ) were conducted to understand the local structure (nearest-neighbor symmetry) of these segregated boundaries.

## DIRECTIONAL SOLIDIFICATION OF EUTECTIC CERAMICS

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The eutectic architecture, a continuous reinforcing phase within a higher volume phase or matrix, can be described as a naturally occurring in-situ composite. The phases comprising a eutectic are thermodynamically compatible at higher homologous temperatures than man-made composites and as such offer the potential for superior high temperature properties and stability.

The mechanical properties of two phase eutectics can be superior to that of either constituent alone due to the strong constraining effects of the interlocking microstructure. The time dependent characteristics of directionally solidified  $\text{Al}_2\text{O}_3/\text{YAG}$  and  $\text{Al}_2\text{O}_3/\text{ZrO}_2(\text{Y}_2\text{O}_3)$  eutectics are presented. The relationships between lifetime, stress and temperature will be discussed. The solidification characteristics of  $\text{Al}_2\text{O}_3/\text{Y}_3\text{Al}_5\text{O}_{12}$  off-eutectic liquid and its relation to achieve in-situ composites materials will be treated at some length. Affects of composition on morphology and on crystallographic orientation are currently topics of investigation in alumina two-phase systems. The effects of varying volume percent of the two constituents and of the addition of dopants have been examined. The approaches to describe and predict high temperature materials processing and properties will be discussed from an experimentalist's point of view.

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